

FORM PTO-1300 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		ATTORNEY'S DOCKET NUMBER: S 4731
		U.S. APPL. NO. (if known, see 37 CFR) 09/856903
INTERNATIONAL APPLICATION NO.: PCT/FR99/02886	INTERNATIONAL FILING DATE: 23 NOVEMBER 1999	PRIORITY DATE CLAIMED: 27 NOVEMBER 1998
TITLE OF INVENTION: NOVEL ALKANOLAMIDE-FREE THICKENING LATEX		
APPLICANT(S) FOR DO/EO/US: Paul MALLO and Guy TABBACHI		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
1. <input checked="" type="checkbox"/>	This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.	
2. <input type="checkbox"/>	This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.	
3. <input checked="" type="checkbox"/>	This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).	
4. <input checked="" type="checkbox"/>	A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.	
5. <input checked="" type="checkbox"/>	A copy of the International Application as filed (35 U.S.C. 371(c)(2))	
6. <input checked="" type="checkbox"/>	a. <input checked="" type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).	
7. <input checked="" type="checkbox"/>	b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. (see attached copy of PCT/IB/308)	
8. <input type="checkbox"/>	c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).	
9. <input checked="" type="checkbox"/>	A translation of the International Application into English (35 U.S.C. 371(c)(2)).	
10. <input type="checkbox"/>	Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).	
11. <input type="checkbox"/>	a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).	
12. <input type="checkbox"/>	b. <input type="checkbox"/> have been transmitted by the International Bureau.	
13. <input type="checkbox"/>	c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.	
14. <input type="checkbox"/>	d. <input type="checkbox"/> have not been made and will not be made.	
15. <input type="checkbox"/>	A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).	
16. <input type="checkbox"/>	An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).	
17. <input type="checkbox"/>	A translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).	
Item 11 to 16, below concern document(s) or information included:		
18. <input checked="" type="checkbox"/>	An Information Disclosure Statement under 37 CFR 1.97 and 1.98.	
19. <input type="checkbox"/>	An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.	
20. <input checked="" type="checkbox"/>	A FIRST preliminary amendment.	
21. <input type="checkbox"/>	A SECOND or SUBSEQUENT preliminary amendment.	
22. <input type="checkbox"/>	A substitute specification.	
23. <input type="checkbox"/>	A change of power of attorney and/or address letter.	
24. <input checked="" type="checkbox"/>	Other items or information:	

International Search Report
 PCT/IB/308
 PCT/IPEA/409
 Application Data Sheet

U.S. APPLICATION NO. 09/856903

INTERNATIONAL APPLICATION NO.
PCT/FR99/02886

ATTORNEY'S DOCKET NO.
S 4731

CALCULATIONS PTO USE ONLY

17. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$ 1,000.00
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$ 860.00
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$ 710.00
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$ 690.00
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$ 100.00

ENTER APPROPRIATE BASIC FEE AMOUNT = \$ 860.00

Surcharge of \$130.00 for furnishing the oath or declaration later than 30 months from the earliest claimed priority date (37 CFR 1.492(e)).

\$ 130.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$
Total claims	12 - 20 =	0	X \$18.00	\$
Independent claims	1 - 3 =	0	X \$80.00	\$
MULTIPLE DEPENDENT CLAIMS(S) (if applicable)			+ \$270.00	\$

TOTAL OF ABOVE CALCULATIONS = \$ 990.00

Reduction of 1/3 for filing by small entity, if applicable. Applicant claims Small Entity Status under 37 CFR 1.27.

+

SUBTOTAL = \$ 990.00

Processing fee of \$130 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.49(f)).

\$

TOTAL NATIONAL FEE = \$ 990.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$

TOTAL FEES ENCLOSED = \$ 990.00

Amount to be
refunded:

charged:

a. ☒ A check in the amount of \$990.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 25-0120 in the amount of \$ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required by 37 CFR 1.16 and 1.17, or credit any overpayment to Deposit Account No. 25-0120. A duplicate copy of this sheet is enclosed.

SEND ALL CORRESPONDENCE TO:

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May 29, 2001

By

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09/856903

JC18 Rec'd PCT/PTC 29 MAY 2001

PATENTS

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Paul MALLO et al.

Serial No. (unknown)

Filed herewith

NOVEL ALKANOLAMIDE-FREE
THICKENING LATEX

PRELIMINARY AMENDMENT

Commissioner for Patents

Washington, D.C. 20231

Sir:

Prior to the first Official Action and calculation of the filing fee, please substitute Claims 1-13 as originally filed, with Claims 1-12 as filed in the Article 34 amendment of December 8, 2000. The pages containing Claims 1-12 are marked "AMENDED SHEET" and are attached hereto. Following the insertion of Claims 1-12, please amend these claims as follows:

IN THE CLAIMS:

Amend claim 3 as follows:

--3. (Amended) Process as defined in claim 1, in which the polymerization reaction is initiated with a redox couple which generates hydrogen sulfite ions (HSO_3^-), such as the cumene hydroperoxide/sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) couple or the cumene hydroperoxide/thionyl chloride (SOCl_2) couple, at a temperature of less than or equal to 10°C , if desired, supplemented with a polymerization coinitiator, such as azobis (isobutyronitrile) (AIBN).

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Paul MALLO et al.

Amend claim 4 as follows:

--4. (Amended) Process as defined in claim 1, characterized in that the anionic polyelectrolyte comprises from 30% to 50% of a monomer comprising a strong acid function and from 70% to 50% either of a monomer comprising a weak acid function or of a neutral monomer.

Amend claim 6 as follows:

--6. (Amended) Process as defined in claim 1, characterized in that the anionic polyelectrolyte is vulcanized and/or branched with a diethylenic or polyethylenic compound in a molar proportion, expressed relative to the monomers used, of from 0.005% to 1% and preferably from 0.01 to 0.1%.

Amend claim 8 as follows:

--8. (Amended) Process as defined in claim 1, characterized in that the oil phase represents from 15% to 40% and preferably from 20% to 25% of its total weight.

Amend claim 9 as follows:

--9. (Amended) Process as defined in claim 1, characterized in that the oil phase consists essentially of isohexadecane or of white mineral oil.

Amend claim 10 as follows:

--10. (Amended) Use of composition obtained according to the process as defined in claim 1, to prepare a cosmetic, dermatopharmaceutical or pharmaceutical topical composition.

Amend claim 11 as follows:

--11. (Amended) Cosmetic, dermatopharmaceutical or pharmaceutical composition comprising from 0.1% to 10% by weight of an inverse latex obtained according to the process as defined in claim 1.--

R E M A R K S

Paul MALLO et al.

The above changes in the claims merely place this national phase application in the same condition as it was during Chapter II of the international phase, with the multiple dependencies being removed. Following entry of this amendment by substitution of the pages, only claims 1-12 remain pending in this application.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE".

Respectfully submitted,

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May 29, 2001

VERSION WITH MARKINGS TO SHOW CHANGES MADE

The claims have been amended as follows:

3. (Amended) Process as defined in ~~either of Claims 1 and 2~~, in which the polymerization reaction is initiated with a redox couple which generates hydrogen sulfite ions (HSO_3^-), such as the cumene hydroperoxide/sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) couple or the cumene hydroperoxide/thionyl chloride (SOCl_2) couple, at a temperature of less than or equal to 10°C , if desired, supplemented with a polymerization coinitiator, such as azobis (isobutyronitrile) (AIBN).

4. (Amended) Process as defined in ~~one of Claims 1 to 3~~, characterized in that the anionic polyelectrolyte comprises from 30% to 50% of a monomer comprising a strong acid function and from 70% to 50% either of a monomer comprising a weak acid function or of a neutral monomer.

6. (Amended) Process as defined in ~~one of Claims 1 to 5~~, characterized in that the anionic polyelectrolyte is vulcanized and/or branched with a diethylenic or polyethylenic compound in a molar proportion, expressed relative to the monomers used, of from 0.005% to 1% and preferably from 0.01 to 0.1%.

8. (Amended) Process as defined in ~~one of Claims 1 to 7~~, characterized in that the oil phase represents from 15% to 40% and preferably from 20% to 25% of its total weight.

9. (Amended) Process as defined in ~~one of Claims 1 to 8~~, characterized in that the oil phase consists essentially of isohexadecane or of white mineral oil.

10. (Amended) Use of composition obtained according to the process as defined in ~~one of Claims 1 to 9~~, to prepare a cosmetic, dermatopharmaceutical or pharmaceutical topical composition.

11. (Amended) Cosmetic, dermatopharmaceutical or pharmaceutical composition comprising from 0.1% to 10% by weight

Paul MALLO et al.

of an inverse latex obtained according to the process as defined
in ~~one of~~ claims 1 ~~to 9~~.

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FO/060, E0695860

Novel alkanolamide-free thickening latex

The present application relates to a novel process for water-in-oil latices, and to the application of these latices as thickeners and/or emulsifiers for skincare products and haircare products or for the manufacture of cosmetic, dermopharmaceutical or pharmaceutical preparations.

Among the various existing thickeners which are used for these purposes, there are, in particular, synthetic thickening polymers, which are in the form of inverse latices, i.e. latices in which the continuous phase is an oil. These latices dissolve extremely quickly; the polymers contained in these inverse latices are, for example, acrylamide/sodium 2-acrylamido-2-methylpropanesulfonate copolymers; they are already naturalized and when they are dissolved in water, for example to a concentration of 1%, it is observed that the pH is generally greater than 6. Such inverse latices are disclosed in the European patent applications published under Nos. EP 0 186 361 and EP 0 503 853. These latices retain considerable thickening capacity even at pH 4.

However, the process for preparing such copolymers uses surfactants of the alkanolamide family, for instance Witcamide™ 511; now, these chemical compounds are liable to degrade into compounds of the nitrosamine family which are themselves products that are known as being potentially carcinogenic. The simple implementation of the precautionary principle thus makes it unavoidable that, in the short or long term, the inverse latices mentioned above will be banned from use in the preparation of cosmetic, dermopharmaceutical or pharmaceutical products. Consequently, the Applicant has become interested in developing a novel process for synthesizing inverse latices which does not have this drawback.

One subject of the invention is a process for preparing a composition in the form of an inverse

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latex, comprising an oil phase, an aqueous phase, at least one emulsifier of water-in-oil (W/O) type, at least one emulsifier of oil-in-water (O/W) type and from 20% to 75% by weight, mainly from 20% to 60% by weight and more particularly from 30% to 45% by weight, of a branched or vulcanized anionic polyelectrolyte, this process successively comprising:

- a step (a) of preparing an aqueous solution containing the monomers and the optional additives;

- a step (b) of emulsifying the aqueous phase prepared in step (a), in an organic phase, in the presence of one or more emulsifiers of water-in-oil type;

- a step (c) of polymerizing the monomers in the aqueous phase, initiated by introducing a free-radical initiator into said phase; and

- a step (d) of adding one or more emulsifiers of oil-in-water type to the resulting dispersion, at a temperature of less than 50°C,

characterized in that:

- the polymerization reaction in step (c) is carried out at a pH of less than 5.5,

- none of said emulsifiers belongs to the alkanolamide family, and

- said anionic polyelectrolyte is based either on a monomer containing a strong acid function, or on at least one monomer containing a strong acid function copolymerized either with at least one monomer containing a weak acid function or with at least one neutral monomer.

According to one variant of this process, the reaction medium obtained from step (b) is concentrated by distillation before carrying out step (c).

According to one preferred embodiment of the process as defined above, the polymerization reaction is initiated with a redox couple which generates hydrogen sulfite ions (HSO_3^-), such as the cumene hydroperoxide/sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) couple, or the cumene hydroperoxide/thionyl chloride (SOCl_2)

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A subject of the invention is, more particularly, a process as defined above, characterized in that 30% to 80% and preferably 30% to 60%, in molar proportions, of the monomer moieties which the anionic

polyelectrolyte comprises contain a strong acid function; and more particularly characterized in that the anionic polyelectrolyte comprises from 30% to 50% of a monomer comprising a strong acid function and from 5 70% to 50% either of a monomer comprising a weak acid function or of a neutral monomer.

The strong acid function in the monomer comprising it is, in particular, the sulfonic acid function or the phosphonic acid function, and said 10 monomer is preferably 2-methyl-2-[(1-oxo-2-propenyl)-amino]-1-propanesulfonic acid. or the acid. The weak acid function in the monomer comprising it is, in particular, the carboxylic acid function, and said monomer is preferably chosen from acrylic acid, 15 methacrylic acid, itaconic acid and maleic acid. The neutral monomer is chosen in particular from acrylamide, 2-hydroxyethyl acrylate, 2,3-dihydroxyethyl acrylate, 2-hydroxyethyl methacrylate, 2,3-dihydroxyethyl methacrylate, or an ethoxylated derivative, with 20 an EO number of between 1 and 20, for each of these esters.

A subject of the invention is, more particularly, a process as defined above, characterized in that the anionic polyelectrolyte comprises, in molar 25 proportions, from 30% to 50% of 2-methyl-2-[(1-oxo-2-propenyl)amino]-1-propanesulfonic [lacuna], partially or totally salified in the form of an alkali metal salt, preferably the sodium salt, or in the form of the ammonium salt, and from 70% to 50% of acrylamide.

30 A subject of the invention is, more particularly, a process as defined above, characterized in that the anionic polyelectrolyte is vulcanized and/or branched with a diethylenic or polyethylenic compound in a molar proportion, expressed relative to 35 the monomers used, of from 0.005% to 1% and preferably from 0.01% to 0.1%, and the vulcanizing agent and/or branching agent is chosen in particular from ethylene glycol methacrylate, sodium diallyloxyacetate, ethylene glycol diacrylate, diallylurea, trimethylolpropane

triacrylate and, more particularly, methylene-bis(acrylamide).

The latex obtained by the process according to the invention generally contains from 2.5% to 15% by weight and preferably from 4% to 9% by weight of emulsifiers, among which from 20% to 50% and in particular from 25% to 40% of the total weight of the emulsifiers present are of the water-in-oil (W/O) type and in which from 80% to 50% and in particular from 75% to 60% of the total weight of the emulsifiers are of the oil-in-water (O/W) type.

According to one particular aspect of the process as defined above, the emulsifiers of the water-in-oil type consist essentially of sorbitan monooleate.

According to another particular aspect, the composition obtained by the process as defined above is characterized in that the oil phase represents from 15% to 40% and preferably from 20% to 25% of its total weight. This oil phase consists either of a commercial mineral oil containing saturated hydrocarbons of paraffinic, isoparaffinic or cycloparaffinic type, having at room temperature a density of between 0.7 and 0.9 and a boiling point of greater than 180°C, such as, for example, Exxsol D 100 S sold by EXXON or a white mineral oil, such as Marcol™ 52, or the isohexadecane sold by Bayer or isododecane, or of a plant oil or of a synthetic oil, or of a mixture of several of these oils.

According to one preferred aspect of the present invention, the composition obtained by the process as defined above is characterized in that the oil phase consists essentially of isohexadecane or of Marcol™ 52. Isohexadecane, which is identified in Chemical Abstracts by the RN number = 93685-80-4, is a mixture of C₁₂, C₁₆ and C₂₀ isoparaffins containing at least 97% C₁₆ isoparaffins, among which the main constituent is 2,2,4,4,6,8,8-heptamethylnonane (RN = 4390-04-9). Marcol™ 52 is a commercial oil corresponding to the definition of liquid petroleum of

the French Codex. This is a white mineral oil in accordance with FDA regulations 21 CFR 172.878 and CFR 178.3620(a) and is registered in the US Pharmacopoeia in US XXIII (1995) and in the European pharmacopoeia (1993).

The latices contain between 20% and 50% water. The latices according to the invention may also contain various additives such as complexing agents, transfer agents or chain-limiting agents.

A subject of the invention is also the use of the composition obtained according to the process as defined above, to prepare a cosmetic, dermo-pharmaceutical or pharmaceutical topical composition.

A topical composition according to the invention, intended to be applied to human or animal skin or mucous membranes, may consist of a topical emulsion comprising at least one aqueous phase and at least one oil phase. This topical emulsion may be of the oil-in-water type. More particularly, this topical emulsion may consist of a fluid emulsion, such as a milk or a fluid gel. The oil phase of the topical emulsion may consist of a mixture of one or more oils.

A topical emulsion according to the invention may be intended for cosmetic use or may be used to prepare a medicinal product intended for treating diseases of the skin and of mucous membranes. In the latter case, the topical composition then comprises an active principle which may consist, for example, of an anti-inflammatory agent, a muscular relaxant, an anti-fungal agent or an antibacterial agent.

When the topical composition is used as a cosmetic composition intended to be applied to the skin or mucous membranes, it may or may not comprise an active principle, for example a moisturizer, a tanning agent, a sunscreen, an anti-wrinkle agent, a slimming agent, a free-radical scavenger, an antiacne agent or an antifungal agent.

A topical composition according to the invention usually comprises between 0.1% and 10% by

weight of the thickener defined above. The pH of the topical composition is preferably greater than or equal to 5 and is more preferably between 6 and 12.

The topical composition may also comprise
5 compounds conventionally included in compositions of this type, for example fragrances, preserving agents, colorants, emollients or surfactants.

According to yet another aspect, the invention relates to the use of the novel thickener in accordance
10 with the invention mentioned above, to thicken and emulsify a topical composition comprising at least one aqueous phase.

The cosmetic, dermopharmaceutical or pharmaceutical composition defined above generally
15 comprises from 0.1% to 10% and more particularly between 0.5% and 5% by weight of said inverse latex. It is in particular in the form of a milk, a lotion, a gel, a cream, a cream-gel, a soap, a bubble bath, a balm, a shampoo or a conditioner.

In general, said inverse latex may advantageously replace the products sold under the name Sepigel™ 305 or Sepigel™ 501 by the Applicant, in cosmetic, dermopharmaceutical or pharmaceutical compositions, since it also has good compatibility with
20 the other excipients used for the preparation of formulations such as milks, lotions, creams, soaps, [lacuna] baths, balms, shampoos and conditioners. It may also be used in combination with said Sepigel products.

It is especially compatible with the concentrates disclosed and claimed in international publications WO 92/06778, WO 95/04592, WO 95/13863, WO 98/47610 and FR 2 734 496, or with the surfactants disclosed in WO 93/08204.

It is particularly compatible with
35 Montanov™ 68, Montanov™ 82, Montanov™ 202 and Sepiperl™ N. It may also be used in emulsions of the type disclosed and claimed in EP 0 629 396 and in cosmetically or physiologically acceptable aqueous

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dispersions with a polyorganosiloxane compound chosen, for example, from those disclosed in WO 93/05762 or in WO 93/21316. It may also be used to form cosmetically or physiologically acceptable aqueous gels of acidic pH, such as those disclosed in WO 93/07856; it may also be used in combination with nonionic celluloses to form, for example, styling gels, such as those disclosed in EP 0 684 024, or alternatively in combination with fatty acid esters of sugars, to form compositions for treating the hair or the skin such as those disclosed in EP 0 603 019, or alternatively in shampoos or conditioners as disclosed and claimed in WO 92/21316, or finally in combination with an anionic homopolymer such as Carbopol™ to form hair treatment products such as those disclosed in DE 195 23596. It is also compatible with many active principles such as, for example, self-tanning agents, for instance dihydroxyacetone (DHA) or antiacne agents; it may thus be introduced into self-tanning compositions such as those claimed in EP 0 715 845, EP 0 604 249, EP 0 576 188 or WO 93/07902. It is also compatible with N-acyl amino acid derivatives, which allows its use in soothing compositions in particular for sensitive skin, such as those disclosed or claimed in WO 92/21318, WO 94/27561 or WO 98/09611. It is also compatible with glycolic acids, with lactic acid, with salicylic acid, retinoids, phenoxyethanol, sugars, glyceraldehyde, xanthans, fruit acids and the various polyols used in the manufacture of cosmetic formulations.

A subject of the invention is thus also the use of an inverse latex as defined above to prepare a cosmetic, dermatopharmaceutical or pharmaceutical composition.

The examples which follow are intended to illustrate the present invention without, however, limiting it.

Example 1: Preparation of the latex according to the invention

a) The following are loaded into a beaker, with stirring

- 5 - 80 g of deionized water,
 - 95.96 g of aqueous 48% (by weight) sodium hydroxide solution,
 - 246.7 g of 2-methyl-2-[(1-oxo-2-propenyl)amino]-1-propanesulfonic acid,
 - 10 - 253.8 g of 50% acrylamide,
 - 0.45 g of sodium diethylenetriaminepentaacetate,
 - 0.132 g of methylenebis(acrylamide);
- the pH of the aqueous phase described above is adjusted to about 5.0 and the amount of aqueous phase is made up
- 15 to 682 g by adding deionized water. In parallel, an organic phase is prepared by introducing the following successively into a stirred beaker:
 - 220 g of isohexadecane,
 - 21 g of MontaneTM 80 VG (sorbitan oleate sold by
 - 20 SEPPIC)
 - 0.2 g of AIBN.

The aqueous phase is gradually introduced into the organic phase and is then subjected to vigorous mechanical stirring using an Ultra-Turrax[®] mixer sold

25 by IKA.

The emulsion obtained is then transferred into a polymerization reactor. The emulsion is bubbled with a strong stream of nitrogen so as to remove the oxygen, and is cooled to about 5-6°C.

30 5 ml of a solution containing 0.28% (by weight) of cumene hydroperoxide in isohexadecane are then added.

After a sufficient amount of time for good homogenization of the solution, an aqueous sodium metabisulfite solution (2.5 g in 100 ml of water) is

35 then added at a rate of 0.5 ml/minute. The addition is carried out over about 60 minutes. During this addition, the temperature in the polymerization reactor

is allowed to rise up to the final polymerization temperature.

The reaction medium is then maintained at this temperature for about 90 minutes.

5 The mixture is cooled to a temperature of about 35°C and 50 g of sorbitan oleate ethoxylated with 20 mol of ethylene oxide are added slowly.

The desired emulsion is obtained:

Evaluation of the properties

10 + viscosity in water containing 2% latex (Brookfield RVT spindle 6, speed 20): 23 450 mPa.s
(Brookfield spindle 6, speed 5): η = 69 000 mPa.s.

15 b) The process described above in paragraph a) is repeated, replacing the isohexadecane with Marcol™ 52, to prepare a latex based on white mineral oil.

The examples which follow use any one of the emulsions prepared in Example 1.

20 **Example 2: Care cream**

	Cyclomethicone:	10%
	Composition 1:	0.8%
	Montanov™ 68:	2%
	Stearyl alcohol:	1%
25	Stearic alcohol:	0.5%
	Preserving agent:	0.65%
	Lysine:	0.025%
	EDTA (disodium salt):	0.05%
	Xanthan gum:	0.2%
30	Glycerol:	3%
	Water:	qs 100%

Example 3: Care cream

	Cyclomethicone:	10%
35	Composition 1:	0.8%
	Montanov™ 68:	2%
	Perfluoropolymethyl isopropyl ether:	0.5%
	Stearyl alcohol:	1%

	Stearic alcohol:	0.5%
	Preserving agent:	0.65%
	Lysine:	0.025%
	EDTA (disodium salt):	0.05%
5	Pemulen™ TR:	0.2%
	Glycerol:	3%
	Water:	qs 100%

Example 4: Aftershave balm

10 FORMULA

	A	Composition 1:	1.5%
		Water:	qs 100%
	B	Micropearl™ M 100:	5.0%
		Sepicide™ CI:	0.50%
15		Fragrance:	0.20%
		95° ethanol:	10.0%

PROCEDURE

Add B to A.

20 **Example 5: Satin body emulsion**

FORMULA

	A	Simulsol™ 165:	5.0%
		Lanol™ 1688:	8.50%
		Karite butter:	2%
25		Liquid paraffin:	6.5%
		Lanol™ 14M:	3%
		Lanol™ S:	0.6%
	B	Water:	66.2%
	C	Micropearl™ M 100:	5%
30	D	Composition 1:	3%
	E	Sepicide™ CI:	0.3%
		Sepicide™ HB:	0.5%
		Monteine™ CA:	1%
		Fragrance:	0.20%
35		Vitamin E acetate	0.20%
		Sodium pyrrolidinone- carboxylate:	1% (moisturizer)

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PROCEDURE

Add C to B, emulsify B in A at 70°C, then add D at 60°C, followed by E at 30°C.

5 **Example 6: Body milk**

FORMULA

10	A	Simulsol™ 165:	5.0%
		Lanol™ 1688:	12.0%
		Lanol™ 14M:	2.0%
		Cetyl alcohol:	0.3%
		Schercemol™ OP:	3%
	B	Water:	qs 100%
	C	Composition 1:	0.35%
	D	Sepicide™ CI:	0.2%
15		Sepicide™ HB:	0.5%
		Fragrance:	0.20%

PROCEDURE

Emulsify B in A at about 75°C; add C at about 60°C, followed by D at about 30°C.

20

Example 7: O/W cream

FORMULA

25	A	Simulsol™ 165:	5.0%
		Lanol™ 1688:	20.0%
		Lanol™ P:	1.0% (additive with stabilizing effect)
	B	Water:	qs 100%
	C	Composition 1:	2.50%
30	D	Sepicide™ CI:	0.20%
		Sepicide™ HB:	0.30%

PROCEDURE

Introduce B into A at about 75°C; add C at about 60°C, followed by D at about 45°C.

35

Example 8: Nongreasy antisen gel

FORMULA

A	Composition 1:	3.00%
	Water:	30%

Example 9: Antisun milk

15	A	Sepiperl™ N:	3.0%
		Sesame oil:	5.0%
		Parsol™ MCX:	5.0%
		λ carrageenan:	0.10%
	B	Water:	qs 100%
20	C	Composition 1:	0.80%
	D	Fragrance:	qs
		Preserving agent:	qs

Emulsify B in A at 75°C and then add C at about 60°C,
25 followed by D at about 30°C, and adjust the pH if
necessary.

30	A	Composition 1:	3.5%
		Water:	20.0%
	B	Colorant:	2 drops/100 g
		Water:	qs
	C	Alcohol:	10%
35		Menthol:	0.10%
	D	Silicone oil:	5.0%

Add B to A; then add C to the mixture, followed by D.

Example 11: Massage care gel

FORMULA

	A	Composition 1:	3.00%
		Water:	30%
5	B	Sepicide™ CI:	0.20%
		Sepicide™ HB:	0.30%
		Fragrance:	0.05%
	C	Colorant:	qs
		Water:	qs 100%
10	D	Micropearl™ SQL:	5.00%
		Lanol™ 1688:	2%

PROCEDURE

Prepare A; add B and then C, followed by D.

15 **Example 12: Radiant-complexion gel**

FORMULA

	A	Composition 1:	4%
		Water:	30%
	B	Elastine HPM:	5.0%
20	C	Micropearl™ M 100:	3%
		Water:	5%
	D	Sepicide™ CL:	0.2%
		Sepicide™ HB:	0.3%
		Fragrance:	0.06%
25		50% sodium pyrrolidinone- carboxylate:	1%
		Water:	qs 100%

PROCEDURE

Prepare A; add B and then C, followed by D.

30

Example 13: Body milk

FORMULA

	A	Sepiperl™ N:	3.0%
		Glyceryl triheptonate:	10.0%
35	B	Water:	qs 100%
	C	Composition 1:	1.0%
	D	Fragrance:	qs
		Preserving agent:	qs

PROCEDURE

Melt A at about 75°C. Emulsify B in A at 75°C and then add C at about 60°C, followed by D.

5 **Example 14: Make-up-removing emulsion containing sweet almond oil**

FORMULA

	Montanov™ 68:	5%
	Sweet almond oil:	5%
10	Water:	qs 100%
	Composition 1:	0.3%
	Glycerol:	5%
	Preserving agent:	0.2%
	Fragrance:	0.3%

15

Example 15: Moisturizing cream for greasy skin

FORMULA

	Montanov™ 68:	5%
	Cetylstearyl octanoate:	8%
20	Octyl palmitate:	2%
	Water:	qs 100%
	Composition 1:	0.6%
	Micropearl™ M100:	3.0%
	Mucopolysaccharides:	5%
25	Sepicide™ HB:	0.8%
	Fragrance:	0.3%

Example 16: Alcohol-free soothing aftershave balm

FORMULA

30	Mixture of lauryl amino acids:	0.1% to 5%
	Magnesium potassium aspartate:	0.002% to 0.5%
	Lanol™ 99:	2%
	Sweet almond oil:	0.5%
	Water:	qs 100%
35	Composition 1:	3%
	Sepicide™ HB:	0.3%
	Sepicide™ CI:	0.2%
	Fragrance:	0.4%

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Example 17: Cream containing AHAs for sensitive skin

FORMULA

	Mixture of lauryl amino acids:	0.1% to 5%
	Magnesium potassium aspartate:	0.002% to 0.5%
5	Lanol TM 99:	2%
	Montanov TM 68:	5.0%
	Water:	qs 100%
	Composition 1:	1.50%
	Gluconic acid:	1.50%
10	Triethylamine:	0.9%
	Sepicide TM HB:	0.3%
	Sepicide TM CI:	0.2%
	Fragrance:	0.4%

Example 18: Aftersun soothing care

FORMULA

	Mixture of lauryl amino acids:	0.1% to 5%
	Magnesium potassium aspartate:	0.002% to 0.5%
	Lanol TM 99:	10.0%
20	Water:	qs 100%
	Composition 1:	2.50%
	Sepicide TM HB:	0.3%
	Sepicide TM CI:	0.2%
	Fragrance:	0.4%
25	Colorant:	0.03%

Example 19: Make-up-removing milk

FORMULA

	Sepiper1 TM N:	3%
30	Primol TM 352:	8.0%
	Sweet almond oil:	2%
	Water:	qs 100%
	Composition 1:	0.8%
	Preserving agent:	0.2%

35

Example 20: Body milk

FORMULA

	Sepiper1 TM N:	3.5%
	Lanol TM 37T:	8.0%

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	Solagum™ L:	0.05%
	Water:	qs 100%
	Benzophenone:	2.0%
	Dimethicone 350 cPs:	0.05%
5	Composition 1:	0.8%
	Preserving agent:	0.2%
	Fragrance:	0.4%

Example 21: Fluid emulsion of alkaline pH

10	Marcol™ 82:	5.0%
	NaOH:	10.0%
	Water:	qs 100%
	Composition 1:	1.5%

Example 22: Fluid foundation

FORMULA

	Simulsol™ 165:	5.0%
	Lanol™ 84D:	8.0%
	Lanol™ 99:	5.0%
20	Water:	qs 100%
	Mineral pigments and fillers:	10.0%
	Composition 1:	1.2%
	Preserving agent:	0.2%
	Fragrance:	0.4%

25

Example 23: Antisun milk

FORMULA

	Sepiperl™ N:	3.5%
	Lanol™ 37T:	10.0%
30	Parsol Nox™:	5.0%
	Eusolex™ 4360:	2.0%
	Water:	qs 100%
	Composition 1:	1.8%
	Preserving agent:	0.2%
35	Fragrance:	0.4%

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Example 24: Eye contour gel

FORMULA

	Composition 1:	2.0%
	Fragrance:	0.06%
5	Sodium pyrrolidinone- carboxylate:	0.2%
	Dow Corning™ 245 fluid:	2.0%
	Water:	qs 100%

10 Example 25: Leave-on care composition

FORMULA

	Composition 1:	1.5%
	Fragrance:	qs
	Preserving agent:	qs
15	Dow Corning™ X2 8360:	5.0%
	Dow Corning™ Q2 1401:	15%
	Water:	qs 100%

Example 26: Slimming gel

20	Composition 1:	5%
	Ethanol:	30%
	Menthol:	0.1%
	Caffeine:	2.5%
	Extract of ruscus:	2%
25	Extract of ivy:	2%
	Sepicide™ HP:	1%
	Water:	qs 100%

Example 27: Alcohol-free soothing aftershave balm

30 FORMULA

A	Lipacide™ PVB:	1.0%
	Lanol™ 99:	2.0%
	Sweet almond oil:	0.5%
B	Composition 1:	3.5%
35 C	Water:	qs 100%
D	Fragrance:	0.4%
	Sepicide™ HB:	0.4%
	Sepicide™ CI:	0.2%

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Example 28: Refreshing aftershave gel

FORMULA

5	A	Lipacide™ PVB:	0.5%
		Lanol™ 99:	5.0%
		Composition 1:	2.5%
	B	Water:	qs 100%
	C	Micropearl™ LM:	0.5%
		Fragrance:	0.2%
		Sepicide™ HB:	0.3%
10		Sepicide™ CI:	0.2%

Example 29: Care for greasy skin

FORMULA

15	A	Micropearl™ M310:	1.0%
		Composition 1:	5.0%
		Octyl isononanoate:	4.0%
	B	Water:	qs 100%
	C	Sepicontrol™ A5:	4.0%
		Fragrance:	0.1%
20		Sepicide™ HB:	0.3%
		Sepicide™ CI:	0.2%
	D	Capigel™ 98:	0.5%
		Water:	10%

Example 30: Cream containing AHAs

FORMULA

30	A	Montanov™ 68:	5.0%
		Lipacide™ PVB:	1.05%
		Lanol™ 99:	10.0%
	B	Water:	qs 100%
		Gluconic acid:	1.5%
		TEA (triethylamine):	0.9%
	C	Composition 1:	1.5%
	D	Fragrance:	0.4%
35		Sepicide™ HB:	0.2%
		Sepicide™ CI:	0.4%

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FORMULA

Example 32: Antisun milk containing Tahitian monoi

FORMULA

Example 33: Antisun care for the face

FORMULA

35 **Example 34: Self-tanning emulsion**

FORMULA

A	Lanol™ 99:	15%
	Montanov™ 68:	5.0%
	Octyl para-methoxycinnamate:	3.0%

B	Water:	qs 100%
	Dihydroxyacetone:	5.0%
	Monosodium phosphate:	0.2%
C	Composition 1:	0.5%
5 D	Fragrance:	0.3%
	Sepicide™ HB:	0.8%
	NaOH:	qs pH = 5

MontanovTM 68 (cetearyl glucoside) is a self-emulsifying
10 composition as disclosed in WO 92/06778, sold by the
company SEPPIC.

Micropearl™ M 100 is an ultrafine powder with a very soft feel and a matt effect, sold by the company Matsumo.

15 Sepicide™ CI, imidazolineurea, is a preserving agent
sold by the company SEPPIC.

PemulenTM TR is an acrylic polymer sold by Goodrich.

Simulsol™ 165 is self-emulsifying glyceryl stearate, sold by the company SEPPIC.

20 Lanol™ 1688 is a nongreasy emollient ester sold by the
company SEPPIC.

Lanol™ 14M and Lanol® S are consistency factors sold by the company SEPPIC.

25 Sepicide™ HB, which is a mixture of phenoxyethanol, methyl paraben, ethyl paraben, propyl paraben and butyl paraben, is a preserving agent sold by the company SEPPIC.

Monteine™ CA is a moisturizer sold by the company SEPPIC.

30 Schercemol™ OP is a nongreasy emollient ester.

Lanol™ P is a stabilizing additive sold by the company SEPPIC.

Parsol™ MCX is octyl para-methoxycinnamate, sold by the company Givaudan.

35 Sepiperl™ N is a nacreous agent sold by the company
SEPPIC, based on a mixture of alkylnpolyglucosides such
as those disclosed in WO 95/13863.

Micropearl™ SQL is a mixture of microparticles containing squalane which is released by the action of massaging; it is sold by the company Matsumo.

Lanol™ 99 is isononyl isononanoate, sold by the company SEPPIC.

Lanol™ 37T is glyceryl triheptanoate, sold by the company SEPPIC.

Solagum™ L is a carrageenan sold by the company SEPPIC.

Marcol™ 82 is a liquid paraffin sold by the company

Esso.

Lanol™ 84D is dioctyl malate, sold by the company SEPPIC.

Parsol Nox™ is a sunscreen sold by the company Givaudan.

Eusolex™ 4360 is a sunscreen sold by the company Merck.

Dow Corning™ 245 fluid is cyclomethicone, sold by the company Dow Corning.

Lipacide™ PVB is a palmitoylated wheat protein hydrolyzate, sold by the company SEPPIC.

Micropearl™ LM is a mixture of squalane, poly(methyl methacrylate) and menthol, sold by the company SEPPIC.

Sepicontrol™ A5 is a mixture of capryloylglycine, sarcosine and extract of Cinnamon zylanicum, sold by the company SEPPIC, such as those disclosed in the

international patent application PCT/FR98/01313 filed on 23 June 1998.

Capigel™ 98 is an acrylate copolymer sold by the company SEPPIC.

Lanol™ 2681 is a mixture of caprylate and coconut caprylate, sold by the company SEPPIC.

Montanov™ 202 is a composition as disclosed in WO 98/47610, sold by the company SEPPIC.

CLAIMS

1. Process for preparing a composition in the form of an inverse latex, comprising an oil phase, an aqueous phase, at least one emulsifier of water-in-oil (W/O) type, at least one emulsifier of oil-in-water (O/W) type and from 20% to 75% by weight, mainly from 20% to 60% by weight and more particularly from 30% to 45% by weight, of a branched or vulcanized anionic polyelectrolyte, this process successively comprising:
- a step (a) of preparing an aqueous solution containing the monomers and the optional additives;
 - a step (b) of emulsifying the aqueous phase prepared in step (a), in an organic phase, in the presence of one or more emulsifiers of water-in-oil type;
 - a step (c) of polymerizing the monomers in the aqueous phase, initiated by introducing a free-radical initiator into said phase; and
 - a step (d) of adding one or more emulsifiers of oil-in-water type to the resulting dispersion, at a temperature of less than 50°C, characterized in that:
 - the polymerization reaction in step (c) is carried out at a pH of less than 5.5,
 - none of said emulsifiers belongs to the alkanolamide family,
 - the emulsifiers of the water-in-oil type used consist essentially of sorbitan monooleate or of sorbitan isostearate,
 - said anionic polyelectrolyte is based either on a monomer containing a strong acid function, or on at least one monomer containing a strong acid function copolymerized either with at least one monomer containing a weak acid function or with at least one neutral monomer, and

- from 30% to 80% and preferably 30% to 60%, in molar proportions, of the monomer moieties which the anionic polyelectrolyte comprises contain a strong acid function.

5 2. Process as defined in Claim 1, in which the reaction medium obtained from step (b) is concentrated by distillation before carrying out step (c).

3. Process as defined in either of Claims 1 and 2, in which the polymerization reaction is initiated with a redox couple which generates hydrogen sulfite ions (HSO_3^-), such as the cumene hydroperoxide/sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) couple or the cumene hydroperoxide/thionyl chloride (SOCl_2) couple, at a temperature of less than or equal to 10°C , if desired, 10 supplemented with a polymerization coinitiator, such as azobis(isobutyronitrile) (AIBN). 15

4. Process as defined in one of Claims 1 to 3, characterized in that the anionic polyelectrolyte comprises from 30% to 50% of a monomer comprising a strong acid function and from 70% to 50% either of a monomer comprising a weak acid function or of a neutral monomer. 20

5. Process as defined in Claim 4, characterized in that the anionic polyelectrolyte comprises, in molar proportions, from 30% to 50% of 2-methyl-2-[(1-oxo-2-propenyl)amino]-1-propanesulfonic [lacuna], partially or totally salified in the form of an alkali metal salt, preferably the sodium salt, or the ammonium salt, and from 70% to 50% either of acrylamide. 25

6. Process as defined in one of Claims 1 to 5, characterized in that the anionic polyelectrolyte is vulcanized and/or branched with a diethylenic or polyethylenic compound in a molar proportion, expressed relative to the monomers used, of from 0.005% to 1% and 30 preferably from 0.01% to 0.1%. 35

7. Process as defined in Claim 6, characterized in that the vulcanizing agent and/or branching agent is

chosen from ethylene glycol methacrylate, sodium diallyloxyacetate, ethylene glycol diacrylate, diallylurea, trimethylolpropane triacrylate and, more particularly, methylenebis(acrylamide).

5 8. Process as defined in one of Claims 1 to 7, characterized in that the oil phase represents from 15% to 40% and preferably from 20% to 25% of its total weight.

9. Process as defined in one of Claims 1 to 8,
10 characterized in that the oil phase consists essentially of isohexadecane or of white mineral oil.

10. Use of the composition obtained according to the process as defined in one of Claims 1 to 9, to prepare a cosmetic, dermopharmaceutical or
15 pharmaceutical topical composition.

11. Cosmetic, dermopharmaceutical or pharmaceutical composition comprising from 0.1% to 10% by weight of an inverse latex obtained according to the process as defined in one of Claims 1 to 9.

20 12. Composition as defined in Claim 11, in the form of a milk, a lotion, a gel, a cream, a soap, a bubble bath, a balm, a shampoo or a conditioner.

ABSTRACT

Process for preparing a composition in the form of an inverse latex, comprising an oil phase, an aqueous phase, at least one emulsifier of water-in-oil (W/O) type, at least one emulsifier of oil-in-water (O/W) type and from 20% to 75% by weight, mainly from 20% to 60% by weight and more particularly from 30% to 45% by weight, of a branched or vulcanized anionic polyelectrolyte, characterized in that the polymerization reaction is carried out at a pH of less than 5.5, in that the emulsifiers do not belong to the alkanolamide family and in that said anionic polyelectrolyte is based either on a monomer containing a strong acid function, or on at least one monomer containing a strong acid function copolymerized either with at least one monomer containing a weak acid function or with at least one neutral monomer. Compositions obtained and cosmetic applications.

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COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

NOVEL ALKANOLAMIDE-FREE THICKENING LATEX

the specification of which: *(check one)*

REGULAR OR DESIGN APPLICATION

- ☐ is attached hereto.
- ☐ was filed on _____ as application Serial No. _____ and was amended on _____ (if applicable).

PCT FILED APPLICATION ENTERING NATIONAL STAGE

- ☒ was described and claimed in International application PCT/FR99/02886 filed on 23 November 1999 and as amended on _____ (if any).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

PRIORITY CLAIM

I hereby claim foreign priority benefits under 35 USC 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN APPLICATION(S)

Country	Application Number	Date of Filing (day, month, year)	Priority Claimed
France	98 14965	27 November 1998	yes

(Complete this part only if this is a continuing application.)

I hereby claim the benefit under 35 USC 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of 35 USC 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status—patented, pending, abandoned)

POWER OF ATTORNEY

The undersigned hereby authorizes the U.S. attorney or agent named herein to accept and follow instructions from L'Air Liquide as to any action to be taken in the Patent and Trademark Office regarding this application without direct communication between the U.S. attorney or agent and the undersigned. In the event of a change in the persons from whom instructions may be taken, the U.S. attorney or agent named herein will be so notified by the undersigned.

As a named inventor, I hereby appoint the registered patent attorneys represented by Customer No. **000466** to prosecute this application and transact all business in the Patent and Trademark Office connected therewith, including: **Robert J. PATCH, Reg. No. 17,355, Andrew J. PATCH, Reg. No. 32,925, Robert F. HARGEST, Reg. No. 25,590, Benoit CASTEL, Reg. No. 35,041, Eric JENSEN, Reg. No. 37,855, Thomas W. PERKINS, Reg. No. 33,027, and Roland E. LONG, Jr., Reg. No. 41,949,**

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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